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ANALYTICAL CHEMISTRY AND SPECTROGRAPHY AS ESSENTIAL CONTROLS FOR DEVELOPMENT AND PRODUCTION IN THE MINERAL AND METAL INDUSTRIES

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ANALYTICAL CHEMISTRY AND SPECTROGRAPHY AS ESSENTIAL CONTROLS FOR DEVELOPMENT AND PRODUCTION IN THE MINERAL AND METAL INDUSTRIES

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1. Development of Analytical Chemistry Instrumentation and Spectrographic Techniques and Apparatus in the Past Two Decades

(a) Prior to World War II the mining and metallurgical industries were able to cope with their analytical problems by means of the slow methods of classical analytical chemistry. There were, however, a number of laboratories in universities and government research institutes where instrumental methods were being investigated. This is true even of emission spectrography and polarography which, before the war, were practically unknown in industrial laboratories.

(b) The enormous demands on production of both well-established and new materials during the war brought sharply to notice the importance of analytical chemistry and the need for analytical control. It is true to say that one major wartime enterprise, involving the production of fissionable material, nearly foundered through initial lack of appreciation of the analytical effort and problem involved.

(c) The tremendous wartime and post-war increase in the number of chemical analyses required, found the western world acutely short of skilled analysts. In order to make up for this shortage, the development of instrumental methods, which were capable of operation by the semi-skilled technician, was greatly accelerated as the only possible solution to this urgent problem.

(d) Novel analytical techniques previously only practised by research workers in universities and research institutes soon became commonplace necessities in the industrial laboratories. In the past two decades widespread adoption has occurred of the polarograph, the spectrophotometer, the flame photometer, the infra-red spectrometer, the direct-recording spectrograph and the X-ray fluorescence spectrograph. Recently the vacuum spectrograph,

the gas chromatograph, the electron probe microanalyser and mass spectrometer have entered industrial research laboratories.

(e) In emission spectrographic analysis alone, the novel techniques of atomic absorption, carrier distillation, arc stabilization, the plasma jet and the application of the hollow cathode have been put to increasing use during the last twenty years.

(f) Analytical chemistry has been extended and enriched by applications and analytical developments of solvent extraction and ion-exchange methods and by the techniques of radiochemistry and isotope analysis.

(g) These new analytical tools have resulted in progress in the mineral and metallurgical industries. In the petroleum industry the advent of the infra-red spectrometer and the gas chromatograph have made possible analyses that previously were well-nigh impossible. New purifying techniques such as zone-refining have produced metals of hitherto unobtainable purity and the need for their analysis has resulted in many refinements of analytical techniques. The metallurgist has thus been provided with alloying constituents of a purity such that the effect of very small traces of impurities on the properties of alloys could be studied for the first time. The opportunity for the microanalysis of minerals and metals has been greatly increased by the introduction in recent years of the electron probe and its application to the identification of microstructure in alloys and minerals, and to the analysis of inclusions in metals and of corrosion products.

(h) It is obvious from the foregoing that the development of mineral resources and metallurgical industries by underdeveloped nations should not be hindered by any great difficulties in the field of analytical chemistry, provided that money is available for the purchase of modern equipment and that trained staff can be obtained. It is beyond the terms of reference of this paper to discuss the financial aspects, but staff training will be considered in a later section.

2. Present Status of Analytical Chemistry in Support of Mineral and Metallurgical Industries

(a) In the economic development of mineral resources from initial prospecting through process development to successful production, analytical chemistry plays an indispensable part. Through the years the improvement in methods of prospecting, the development of new and more efficient processes for winning metals from their ores and the production of high quality metals and alloys of constant composition, have been paced by advances in analytical chemistry.

(b) Prospecting

(i) The first phase of prospecting is the detection of valuable minerals. The modern prospector has available a number of physical and chemical aids. For example, small portable chemical kits have been designed which use simple methods for the estimation of valuable elements in water, vegetation and rocks. In the geochemical laboratory semi-quantitative results by spectrographic and chemical techniques permit the examination of a large number of samples in a relatively short time. Mobile laboratories have been developed that can be fitted with modern chemical instruments including the emission spectrograph, flame photometers and spectrophotometers. With such equipment available it is possible to investigate an area much more rapidly than in the past. All these facilities enable modern prospecting teams to select more readily the particular mineralized locations that hold promise of becoming mines.

(ii) When the potentially valuable areas are localized, and more intensive exploratory work is undertaken, more accurate analyses become necessary. Economically valuable metals, such as gold and platinum, rarely occur as high grade ores and very accurate trace analysis is necessary in the evaluation of their occurrence. When it is realized that one ounce of gold per ton of ore, or three one-thousandth of one per cent constitutes a rich ore, and one-fifth of that amount may be an economic ore, the necessity for accurate assays in the prospecting phase can be appreciated. Other elements such as platinum and rhodium normally occur in ores in even lower concentrations. Similar requirements arise with a number of strategic metals such as molybdenum, tungsten, niobium and uranium, which normally occur in the range of 0.1-2.0%. The list of elements of economic interest and value is steadily increasing; recently lithium, caesium, beryllium, germanium, niobium, zirconium, uranium and rare earths have all advanced from the status of purely academic interest to being materials of economic importance. Due to their chemical properties, these metals are difficult and tedious to determine in their ores by chemical methods. Instrumental methods are therefore necessary to make rapid and accurate determinations of these elements.

(c) Process Development

(i) When it has been established that ore occurs in sufficient quantity and grade for establishing a mine, the selection of the most efficient methods of beneficiating and otherwise processing the ore is undertaken.

(ii) In process development, particularly for complex ores, the knowledge and skill of the analytical chemist is at a premium. He is presented with compounds where the elements may be in unusual associations and in ratios that make separations difficult. Furthermore, due to the associations, segregation, resulting from gravity differences, may be a complicating factor.

(iii) All techniques and instruments in the field of inorganic analytical chemistry are required to support research in mineral processing, mineralogy and metallurgy. Emission spectrography, X-ray diffraction, X-ray fluorescence spectrometry and electron probe microanalysis are employed to reveal the distribution of the minerals contained in an ore and of the elements within the mineral grains. Wet chemical methods, electrometric techniques, flame photometry, spectrophotometry and fire assaying methods, in addition to those above, are used for the specific accurate assays required.

(iv) Although beneficiation of the ore is usually a physical treatment such as flotation or magnetic separation, the assessment of the efficiency of the operation depends upon the analytical laboratory. Not only should the analyst be competent and ingenious, but he should have an appreciation of the problems of the mining and metallurgical engineer. In this way he will not only provide the assays requested, but be in a position to suggest and perform other assays that might assist with the elucidation of the problem. In effect, when the ore-dressing specialist and analytical chemist are working as an understanding and integrated team, the best conditions for research are achieved.

(v) Pyrometallurgical processes for the reduction to metal, purification by liquid-liquid extraction in the uranium industry, pressure leaching, ion-exchange as used in rare earth separations, and distillation procedures for the separation and purification of volatile metals, all require analytical control to ensure efficient operation. Observing the increasing number of processes involved in modern metallurgy, and the wide variety of products, it is readily seen that the analytical services must be versatile. This is particularly true in the development stage. The premium on high-quality products makes it imperative that the process development be directed to this end. Conversely the "penalty metal" clause can render uneconomic a process that fails to remove the undesired impurity. Control of these aspects of process development is a function of the analytical chemist.

(d) Production

(i) Once an economic process has been developed its application to production requires continued analytical control. The production analyst may not have to contend with the wide variations in composition of materials that face his colleague in the development field, but the crude ores or concentrates for the production process may still, unavoidably, be far from constant in composition and require careful, accurate analysis to guide the process engineers in the modifications necessary to treat raw materials of varying composition.

(ii) The product is normally held to close quality tolerances and it is the analyst's responsibility to demonstrate that these requirements are being maintained by the plant. The fact that very little compositional variation is permitted in the product, permits the application of automation in analytical

control. For the production of materials such as ferrous and non-ferrous alloys, control is increasingly exercised by automatic instruments such as the direct recording emission- and X-ray spectrograph, and radiochemical methods are used in some analyses of very high purity materials. Normal methods of analysis are time-consuming and the application of rapid automatic methods of analysis can result in appreciable saving of time, and hence considerable savings in production costs. Process control concerned with changes in chemical concentration or acidity is clearly an extension of analysis from the laboratory into the plant, and in many plants such controls are indeed manned by staff from the analytical laboratory. Here also there is scope for automation to produce savings in the labour force and to result in less compositional variation in process streams than under manual operation. Although in-stream analysis has been developed largely by the chemical industries, particularly by those producing petroleum, heavy chemicals and foods, some instruments have been designed to do similar jobs, for the processing of ores. Siggia[1], in his recent book, reviews the field of continuous analysis applied to chemical processes and illustrates the use of X-ray spectrometry to monitor the quality of filter cake from ore processing. In setting up new plants in less developed countries, it should be worthwhile to consider the integrating of continuous in-stream analytical procedures. The advantages of instantaneous data are obvious and such systems are much cheaper when installed concurrently with the fabrication of the plant.

(iii) The general trend toward higher purity requirements for many materials, notably semiconductor ingredients, has made highly accurate analytical control of paramount importance. Even with common materials such as steel, cement and the like, it has been found that minute amounts of certain elements can profoundly affect the properties of the material, and the analyst is increasingly called on for routine analysis in the parts per million or even parts per billion range.

(iv) For such work, many ingenious refinements of emission spectrographic techniques have been developed, for the standard of purity demanded is so high that ordinary chemical reagents cannot be used because of their relative impurity. Recourse must therefore be taken to a method, such as emission spectrography, where little other than very high purity graphite is brought in contact with the material under examination. The spectrographic technique is, apart from the still uncommon methods of radioactivation analysis and solid-source mass spectrometry, the only means of determining impurities at these very low levels.

(v) The production analytical staff together with the production engineers are concerned with trouble shooting. Considerable analytical research may be required to discover causes for alterations in the grade, colour and other properties of the product. It is the function of the production staff to investigate immediate possibilities before referring the problem to the research laboratory.

(vi) The analytical laboratory in the plant is commonly required to provide services for the health and safety of employees. This usually involves determination of levels of toxic substances such as fumes, dust, etc., which result from the different stages of the process.

(vii) From the foregoing it is evident that the analytical laboratory may be required to perform a wide variety of analyses in the mineral and metal processing fields. In less well-developed areas, consulting and referee laboratories will probably not be readily available. Therefore, when planning analytical services, self-sufficiency must be emphasized so that production will not suffer from inadequacy of facilities to meet most contingencies.

3. Staff Requirements for Analytical Laboratories

(a) The staffing of a large or medium sized analytical laboratory in a mining and metallurgical industry in Canada will be described. Suggestions will be advanced as to how a similar laboratory in a less developed area might obtain the necessary personnel.

(b) In analytical chemistry laboratories serving development and production in Canada, the chief chemist usually is a university graduate with five or more years of experience following graduation, or has equivalent qualifications. It is desirable that the development analyst have, in addition, a higher degree, for originality and scientific leadership are needed to ensure that the analytical contribution to the overall research and development problem is adequate in all respects. The chief analyst should be the severest critic of the work put out by his laboratory and should instil in his staff those standards of scientific integrity without which confidence in the results of the analyst is quickly lost.

(c) In addition to a highly qualified chief analyst a number of university graduates, usually acting as project or group leaders, are also required. Most of the routine analytical work is performed by technicians whose training has been gained in one of two ways.

(i) One group completes secondary school and then attends a technical institute for a two or three year course in chemistry. These are well trained and, with experience, become competent analysts. The theoretical subjects included in their curriculum are usually sufficient to allow them to proceed to semi-professional status and in fact be classed as technologists.

(ii) The other category enters the laboratory directly from secondary school, in some cases without completing the course. Many of these, with several years training, become good routine analysts, capable of doing a wide variety of procedures. They are often most useful in instrumental analytical techniques. It must be realized, however, that certain of these, due to lack of ability, never become more than semi-skilled, tied to simple repetitive functions.

(iii) It, perhaps. should be again emphasized that automation and in-stream analyses allow a plant to function with a greatly reduced number of analysts. However, the analytical chemist, not the production engineer, is considered to be responsible for such equipment. This aspect is discussed thoroughly by Siggia[1].

(iv) In the underdeveloped areas, there are then, broadly, the two situations. Firstly, the provision of the highly qualified and experienced leaders of the analytical teams, and secondly, the training of unskilled locally recruited junior staff to provide the technical support.

a If the educational system of the countries concerned includes secondary schools, technical institutes and universities with adequate science faculties, no serious problem exists. Presumably these schools and universities could provide the basic training, and the necessary experience could be obtained in laboratories at home or abroad, as under the present Colombo plan.

b However many of these underdeveloped areas are deficient in centres of higher scientific learning. Persons desiring a career in science or engineering must go abroad to gain the academic qualifications and necessary experience to fit them for senior positions in analytical chemistry. Although this means of providing well-qualified chemists has some advantages, particularly with respect to gaining experience, it does not keep up with the demands of the growing industries. Rather exceptional talents and adaptability are required on the part of the individual, since he must become fluent in the tongue used in the country in which he studies, as well as devote his efforts to his scientific training.

c The alternative is that, for an initial period, highly qualified and experienced expert analysts be loaned to the underdeveloped areas, each expert serving for about three years. Their duty would be to guide the setting up of suitable analytical laboratories, and to advise on the appropriate analytical methods with respect to operation by locally recruited staff. In many ways the most important duty of all, is to start a training scheme for such staff. In this way, the particular industrial development would be supplied quite rapidly with a nucleus of reasonably skilled analytical workers. At the end of his term the foreign expert would select, from the staff he has trained, those whom he considers would benefit from a two- or three-year period of advanced training in similar laboratories of his own land. It is recommended that the majority of the advanced training be in the land of the tutor, for inevitably he will have given his trainees their preliminary instruction in the methods and on the equipment with which he is most familiar. In addition he can also ensure that they will get their further experience under the most advantageous conditions.

d From our observation, the practice of individuals being sent by the authorities of underdeveloped areas to gain further experience abroad does not, in all cases, result in a choice of the laboratories best suited to give the particular training required. In contrast, the foreign expert, who has seen and appreciated the peculiar problems of the particular underdeveloped area, is in a position to know which of the training grounds in his own country will give the trainee the experience of greatest value.

LITERATURE REFERENCE

1. Siggia, Sydney. Continuous Analysis of Chemical Process Systems. John Wiley and Sons Inc., New York, 1959.

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